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ALKALOIDS OF THE EPIGEAL PART OF *Convolvulus krauseanus*

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The epigeal part of *Convolvulus krauseanus* collected in two vegetation periods — flowering and fruit-bearing — has been studied for the first time. Five bases have been isolated. Convolvine, convolamine, and convolidine proved to be known, phyllalbine was isolated from this species for the first time, and colvolicine proved to be a new base for which the structure of (±)-N-acetoxy-3α-veratroylnortropine has been established.

Three alkaloids (convolvine, convolamine, and convolidine) have been isolated previously from the roots of *Convolvulus krauseanus* Rgl. et Schmalh. collected at the end of of the vegetation periods in the environs of the village of Bakhmal (Turkestan range) [1]. We have studied the total bases of the epigeal part of this plant collected in the same place in two vegetation periods. The total alkaloids were isolated by chloroform extraction of the plant moistened with 8% ammonia solution. It was found that the epigeal part collected in the vegetation period (June 3, 1978) contained 0.82% of total alkaloids, and that collected in the fruit-bearing period (August 13, 1978) contained 0.16%. The combined materials obtained were separated into phenolic and nonphenolic fractions, and the latter were separated with citrate-phosphate buffer solutions having pH 6.8 and 5.6. Convolvine with a small amount of convolamine was extracted mainly into the buffer with pH 6.8, and convolamine with traces of other bases into the buffer with pH 5.6. The mother solution contained practically no convolvine or convolamine.

The combined phenolic bases were treated successively with acetone and methanol. Crystals deposited with mp 209-210°C (chloroform—methanol), M^+ 291. A comparison of the IR, mass, and NMR spectra of the base obtained with the spectra of the known alkaloid phyllalbine, which was first isolated from *Phyllanthus discoides* [2], and also a mixed melting point showed their identity. Thus, this is the first time that phyllalbine has been isolated from this species of *Convolvulus*.

After the removal of the phyllalbine, the mother liquor was treated with methanol. The insoluble part was filtered off, and it proved to be a mixture of two bases. They were separated by fractional crystallization. One of the bases was phyllalbine, and the other, with mp 214-215°C, was convolidine, which was first isolated from the roots of this plant.

The mother liquor from the combined alkaloids (from the plant collected in the fruit-bearing period) after the maximum elimination of colvolvine and convolamine was deposited on a column of silica gel and eluted successively with petroleum ether (70-100°C), hexane, ether, chloroform, and a mixture of chloroform and methanol. From the ethereal fractions, a base crystallized with mp 184-185°C, R_f 0.85 (system 1: chloroform—methanol—NH_4OH (8:2:0.1)). The subsequent ethereal fractions yielded a white crystalline substance with mp 144-145°C

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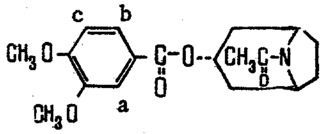
(acetone), R_f 0.82 (system 1), $C_{18}H_{23}NO_5$. In its composition, spectral characteristics, and chemical properties, it differed from bases described previously, and we have called it convolicine (I). According to its IR spectrum (1700 cm^{-1}) the alkaloid contains an aromatic ester carbonyl group, an amide carbonyl (1640 cm^{-1}), and a 1,2,4-trisubstituted benzene ring (800, 810, 880 cm^{-1}). The NMR spectrum confirmed substitution in the aromatic moiety (H_c appeared at δ 6.86 ppm, doublet, J_{ortho} = 8 Hz, and the H_a and H_b signals were in the 7.45-7.65 ppm interval). The signal from the $C_{3\alpha}H$ appeared at 5.32 ppm (1 H, t), that of two aromatic OCH_3 groups at 3.88 ppm (6 H, s), and the protons of a $-N-C(=O)-CH_3$ group at 2.05 ppm (6 H, s).

The mass spectrum contained the peak of the molecular ion with m/e 333 and a peak with m/e 290 (M − 43) and also the peaks of ions with m/e 182, 165, 151, 126, 123, 110, 97, and 95, which are characteristic for alkaloids of the tropane series.

The composition and spectral characteristics permitted the conclusion that convolicine is based on a tropane sleketon, and the subsequent chemical transformations of (I) confirmed this hypothesis.

The alkaline hydrolysis of the alkaloid yielded an acid which was shown by its R_f value and a mixed melting point test to be veratric acid, and the amino alcohol moiety yielded a substance of basic nature with R_f 0.87 (system 2: cyclohexane–chloroform–diethylamine (5:4:1)) and mp 115-116°C (acetone). The mass spectrum of the hydrolysis product gave the peak of the molecular ion with m/e 169, a peak with m/e 125 (M − 43), and the peaks of a tropane ring. The IR spectrum retained the band of an amide carbonyl. Consequently, the hydrolysis of (I) cleaved only an ester bond, and this led to veratric acid and the amino alcohol N-acetoxynortropine. On this basis, we assumed that convolicine is the ester of N-acetoxynortropine and veratric acid. In actual fact, when convolvine was acetylated with acetic anhydride we obtained a substance with mp 144-145°C which was identical with convolicine. Thus, (I) has the structure of (±)-N-acetoxy-3α-veratroylnortropine.



EXPERIMENTAL

Hydrolysis of Convolicine. A mixture of 100 mg of the base and 10 ml of a 5% solution of KOH in methanol was boiled on the water bath for 3 h. The methanol was driven off from the reaction mixture in vacuum. The dry residue was heated on the water bath with ether (5 × 40 ml). The ethereal extracts were combined, dried with anhydrous potassium carbonate, and concentrated. White crystals deposited with mp 115-116°C (acetone).

Acetylation of Convolvine. A mixture of 20 mg of convolvine and 0.5 ml of acetic anhydride was left overnight. Then the excess of acetic anhydride was driven off in vacuum. The residue consisted of a crystalline substance with mp 144-145°C. It gave no depression of the melting point with convolicine.

SUMMARY

The epigeal part of *Convolvulus krauseanus* collected in two vegetation periods — flowering and fruit-bearing — has been studied for the first time. Five bases have been isolated: the known colvolvine, convolamine, and convolidine; phyllalbine, isolated from this species for the first time; and colvolicine, a new base for which the structure has been established.

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